LA-UR-21-20541

Title: An Investigation of Critical Pit Size in SAVY 4000 Wall

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Intended for: Report

Issued: 2021-01-21

An Investigation of Critical Pit Size in SAVY 4000 Wall

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1. Introduction

The Department of Energy (DOE) issued DOE M 441.1-1, Nuclear Material Packaging Manual, in March 2008 to protect workers who handle nuclear material from exposure due to loss of containment of stored materials. The Manual specifies a detailed approach to achieve high confidence in containers and includes requirements for container design and performance, design-life determinations, material contents, and surveillance and maintenance to ensure container integrity over time. Nuclear Filter Technology, Inc. (NFT Inc.), with the oversight of Los Alamos National Laboratory (LANL), designed the SAVY 4000 container as a simple, robust and reusable container for storing nuclear materials [1][2]. The design of this container includes a filter to prevent pressurization and to facilitate the release of hydrogen, thus preventing flammable gas mixtures from forming. The container is comprised of a corrosion resistant 316L stainless steel containment boundary, ceramic filter for the prevention of radiological particulate release, and a chemically stable O-ring made of Viton®. The SAVY 4000 lifetime was extended in July of 2019 from 5 years to 15 years [3]. The desired lifetime for the SAVY 4000 is 40 years, but due to corrosion and the affect it may have on accident performance the lifetime could not be extended beyond 15 years. A study was designed to investigate the affect that pit corrosion has in a drop scenario to define the maximum pit size that could be present without compromising the SAVY's ability to retain its contents [4]. In order to better understand eh true design life of the SAVY 4000 series of containers it is vital to recognize the size a pit needs to be to compromise the container in a drop scenario and to relate that pit size to the corrosion rates seen during storage.

2. Experimental

The critical pit size study consisted of five phases; micro-tensile tests with pitted 316 stainless steel foils to obtain degraded mechanical properties, finite element analysis using degraded material properties, corroding of SAVY sections to determine exposure durations and solution concentrations, and finally a verification drop test of a whole pitted container. Each phase is described in the following subsections.

2.1 Micro-tensile testing of corroded foils

Grade 316 stainless steel foils were exposed to a ferric chloride solution with varying concentrations and exposure times. The foils used had an initial dimension of 100 mm x 100 mm x 0.1 mm. Ferric chloride solutions of 1.5M and 3M were used. The 1.5M solution was exposed to the foil for 10 minutes while the 3M solution was exposed to the foil for 10 minutes and 20 minutes (nomenclature shown in Table 1), all exposures were done at ambient temperature. It is worth noting that at 20 minutes, the 1.5M solution perforated the foils and was omitted for testing.

TABLE 1 CROSSWALK OF NOMENCLATURE

		Exposure Times	
		10 minutes	20 minutes
Solution Concentrations	1.5M	$1.5M_{10}$	N/A
	3.0M	$3M_{10}$	$3M_{20}$

After exposure to the corrosive environment the foils were measured by optical profilometry using a GoCator 2520 profiler in order to characterize the pit development. Dog bones seen in Figure 1 were laser machined out of the pitted foils with the following dimensions: 0.4 mm (length) x 0.085 mm (width) x 0.1 mm (thickness). The machined dog bones were pulled on a lab built micro-tensile test apparatus using a piezo flexure-guided actuator made by Physik Instrument. The maximum load the actuator was capable of pulling was 10 N with a 0.1 mN resolution. The maximum displacement that the actuator was able to achieve was 0.5 mm with a 25 nm resolution. The output load and displacement behavior provided the degraded mechanical properties in terms of tensile stresses (yield strength and ultimate tensile strength) and elongations (uniform and total elongation). Five categories were designated for the micro-tensile testing: as-received, $1.5M_{10}$ small pit, $1.5M_{10}$ large pit, $3M_{10}$ and $3M_{20}$.

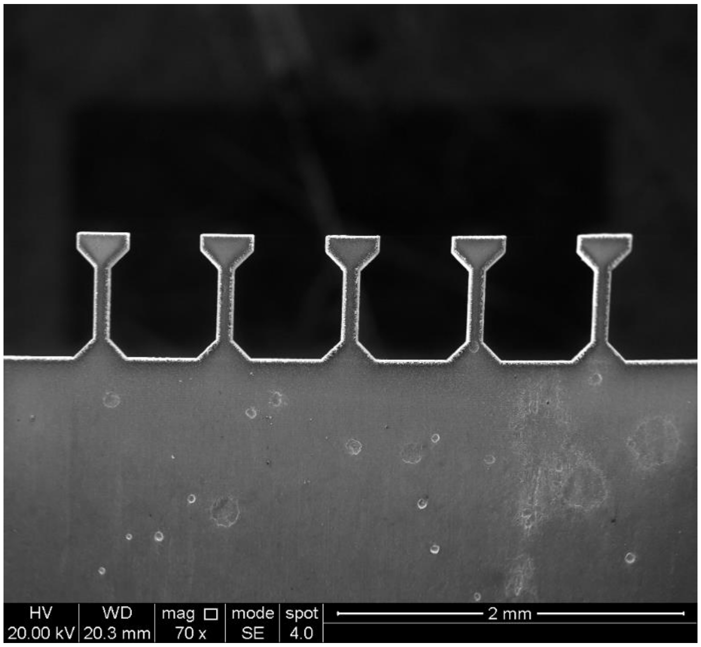


FIGURE 1 IMAGE OF DOG BONES MACHINED IN 316 SS FOIL.

2.2 Finite element analysis with degraded properties

Using the data collected in section 2.1 a finite element model was generated using Abaqus/Eplicit 2019 to predict the behavior of the container in a drop scenario. A prediction was made on the level of pitting that would be required to achieve the maximum amount of stress within the container body without causing a rupture. The result of the FEA would then be compared to the true pitted container drop result in order to validate the model.

2.3 Corroded SAVY 4000 sections

The intent of corroding SAVY sections before the whole container was to gain an understanding of how long a corrosive solution would need to be in contact with a container body in order to achieve a particular pit size. This phase of the study was an iterative process to narrow in on the desired pit size.

Using 3.0 and 1.5 molar solutions of $FeCl_3$, the SAVY body sections were exposed for a variety of times. The sections were then examined using an optical profilometer to determine the resulting pit sizes based on the exposure times that the sections were exposed to. Once the desired pit size was achieved the solution concentration and exposure duration was applied to the whole SAVY container.

2.4 Pitting whole SAVY

In order to achieve the most complete coverage that did not prematurely compromise the containers integrity, 30 mL of 1.5 molar $FeCl_3$ solution was applied in strips that ran parallel to the vertical axis without overlapping any previous exposures. The container was placed on a stand to hold the container in a horizontal position over the duration of the exposure time (100 minutes) for each strip until the inner surfaces were fully corroded, except for the small areas between exposure strips. After each exposure, the $FeCl_3$ solution was removed and the container was rinsed with DI water to stop the corrosion. Once all sides had been corroded the bottom of the container body was exposed to 50 mL of 1.5 molar $FeCl_3$ solution for 100 minutes. The $FeCl_3$ solution was then removed and the container rinsed and dried. To validate the pit sizes generated in the container, the eddy current measurement device included in the Modular Non-destructive Test System (MINTS) was employed to measure the defects [5].

2.5 Drop testing of pitted SAVY

The bases for a failing drop test or a passing drop test will be a comparison of the pre and post helium leak rates. A pre-drop test helium leak test was performed on a LACO Flexstation™ bell-jar helium mass-spectroscopy leakage tester. The container had to be leak tested inside of PF-4 due to the system outside of TA-55 being inoperable. The drawback to this arrangement was that the container could not be fully assembled for the drop test, forcing the container to be opened and closed once more before performing the drop test.

The drop test was performed in a drop tower that is equipped with an essentially unyielding floor. The drop tower is capable of dropping from a maximum height of 15 feet 8 inches using a swing away drop leaf or ~13 feet 8 inches using a sling system with a pneumatic release (Figure 2). The drop tower was designed to allow containers loaded with a surrogate material (e.g., cerium oxide) to be dropped and have the particulate release measured using aerosol particle counters. The test performed on the pitted 5 quart SAVY container did not contain any surrogate material, and the test was setup to mimic the qualification drop test, e.g. drop orientation, gross weight and height.

The degraded container was dropped from 12 feet using the sling system with pneumatic release. The container's gross weight was 18.3742 kg and was drop center of gravity (CG) over bottom corner (Figure 2). The orientation was chosen to maximize plastic deformations on the container body thus inducing the highest amount of stresses in the container body.

FIGURE 2 DEGRADED CONTAINER HANGING IN FINAL DROP ORIENTATION FROM SLING SYSTEM

3. Results

3.1 Micro-tensile testing results of corroded foils

The profilometry scans showed that the foils developed pits with varying sizes respective of the conditions that they were exposed to (Figure 3). It is seen in (Figure 4) that the depths increase depending on the concentration and exposure times, but the pit diameters are limited to around ~400 µm. It is worth noting that the pit depths with the lower concentration of $FeCl_3$ solution are deeper than the 3M concentration over the same exposure times.

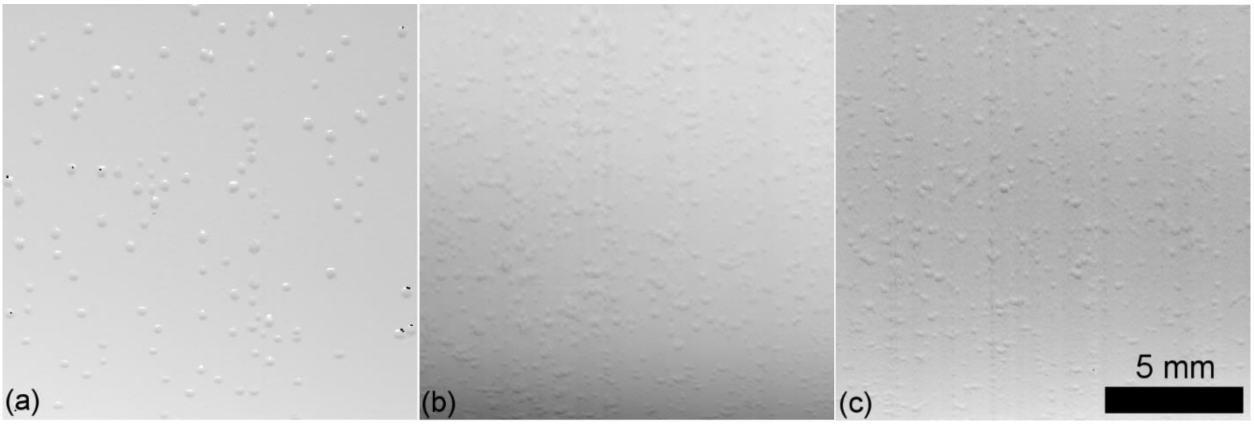


Figure 3 Optical profiler maps of foils after being exposed to $FeCl_3$ of varying concentrations and durations. (a) Foil exposed for 10 minutes to a 1.5M solution. (b) Foil exposed for 10 minutes to a 3.0M solution. (c) Foil exposed for 20 minutes to a 3.0M solution.

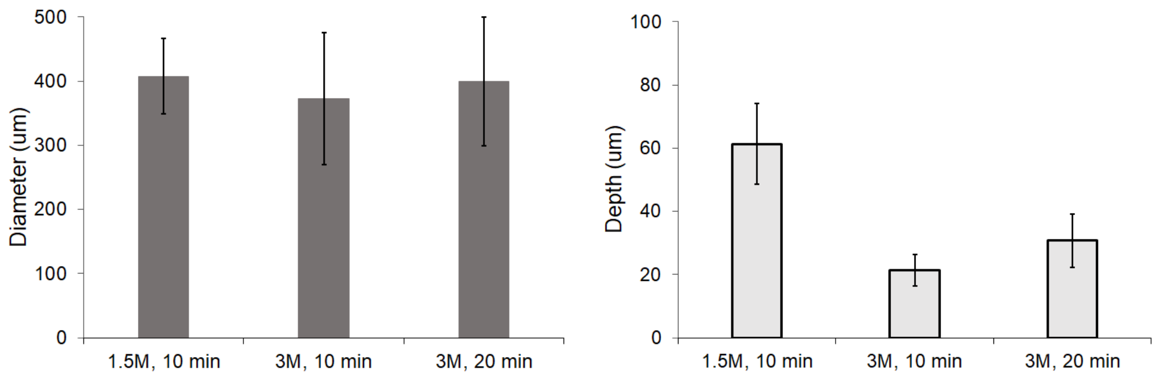


Figure 4 Diameter and depth data from the GoCator 2520 profiler for the respective exposure conditions

A comparison of mechanical properties was made between the as-received foils and the regions between the observed pits in the foils that were exposed to $FeCl_3$ to determine whether there were any effects beyond the expected degradation in the pitted regions. No significant difference, within 10%, was observed between the as-received foils and the areas in the exposed foils that did not have any pitting as is seen in Figure 5.

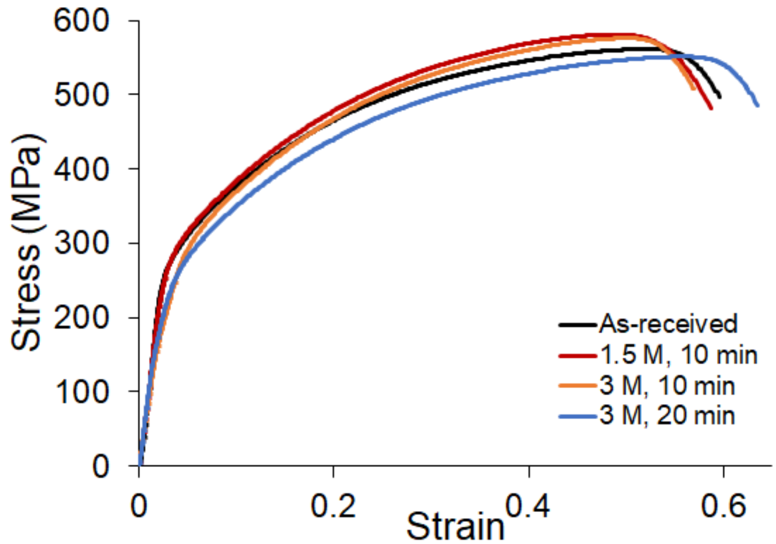


FIGURE 5 STRESS STRAIN CURVES FOR AS-RECEIVED, $1.5M_{10}$, $3.0M_{10}$ AND $3.0M_{20}$ FOILS COLLECTED IN THE MICRO-TENSILE TEST RIG.

Targeted tensile testing was completed on the regions with pits. The pits in the gauge section were larger than the width in most cases. The foils exposed to a $1.5M_{10}$ had to be tensile tested in two categories; small pits and large pits. As can be seen in Figure 6 the 3M solution has a lesser effect on the mechanical properties than the 1.5M solution. The properties from the $1.5M_{10}$ large pit foil sections were severely degraded, 87.27% reduction of the ultimate tensile stress (UTS), due to pits penetrating approximately 70% through the thickness of the foil. The $3.0M_{10}$ had the smallest effect on the mechanical properties with a reduction of approximately 3.0% relative to the as-received foil UTS.

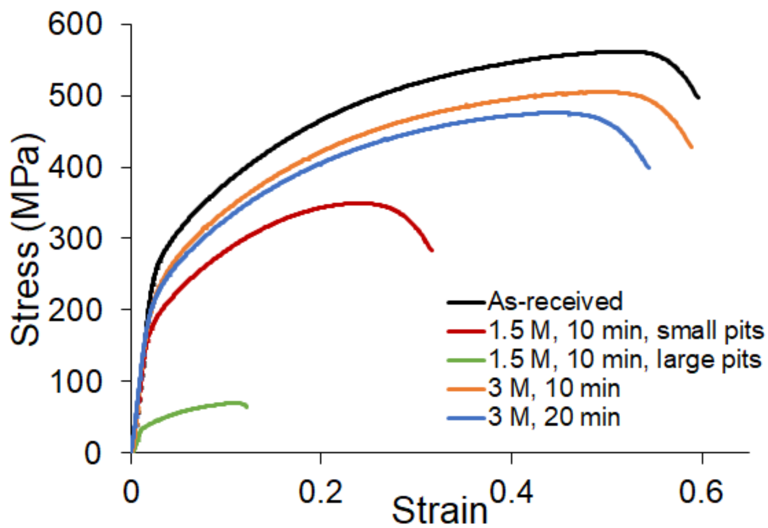


FIGURE 6 STRESS STRAIN CURVES FOR AS-RECEIVED FOILS COMPARED TO THE FOILS WITH PITS IN THEM.

From the foil micro-tensile testing that was completed it was decided that the whole container pit size target should be a pit that is approximately 50% through the total thickness of the container body in order to reduce the UTS enough to approach a failing drop test.

3.2 Finite element analysis results with degraded properties

Using the load – displacement data for the $1.5M_{10}$ small pits collected in the first phase of this study, finite element models were created. The properties used for the model were approximately 38% of the as-received UTS. The results of the FEA showed that the stress would approach but not exceed the UTS of the degraded material. The deformations predicted by the FEA model using the degraded mechanical properties are significantly more than were seen in the qualification drop test (Figure 6).

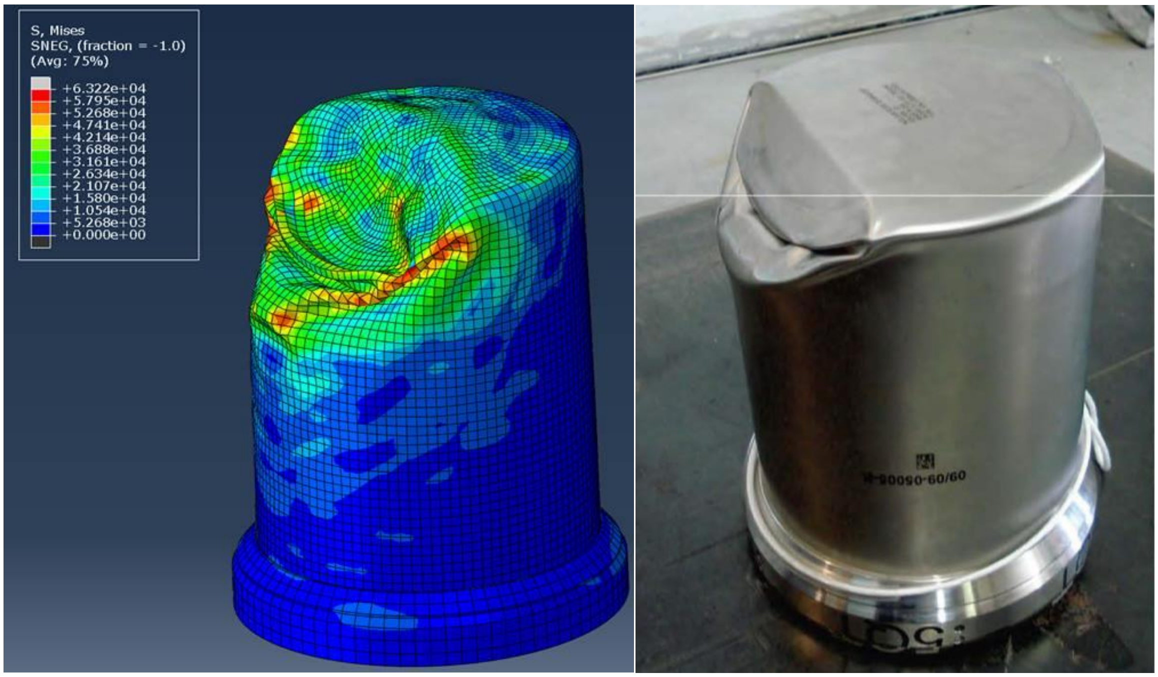


FIGURE 7 COMPARISON OF DEFORMATIONS WITH FEA RESULTS USING DEGRADED MECHANICAL PROPERTIES AND THE ACTUAL DROP TEST RESULT OF A PRISTINE 5 QUART USED IN THE QUALIFICATION DROP TESTING.

3.3 Corroded SAVY 4000 sections results

The profilometry of the pitted SAVY sections showed that the 1.5 M solution would need to be exposed to the SAVY wall for 100 minutes to achieve the desired 50% depth pit.

3.4 Result of pitting whole SAVY

The SAVY that was corroded was picked up directly from the TA-55 warehouse. While the first container was being corroded, a portion of a previously exposed area was overlapped by the exposure that was underway and a pit grew to the point of penetrating the wall (Figure 8). A new container was obtained from the warehouse so that the process could be restarted with a slightly greater spacing between each exposure. Once the container body inner walls were completely corroded (Figure 9) the eddy current instrument on the MINTS was used to measure the pit depths. The MINTS showed that the pits were between 35% and 55%, confirming that the container was in the range that was desired.

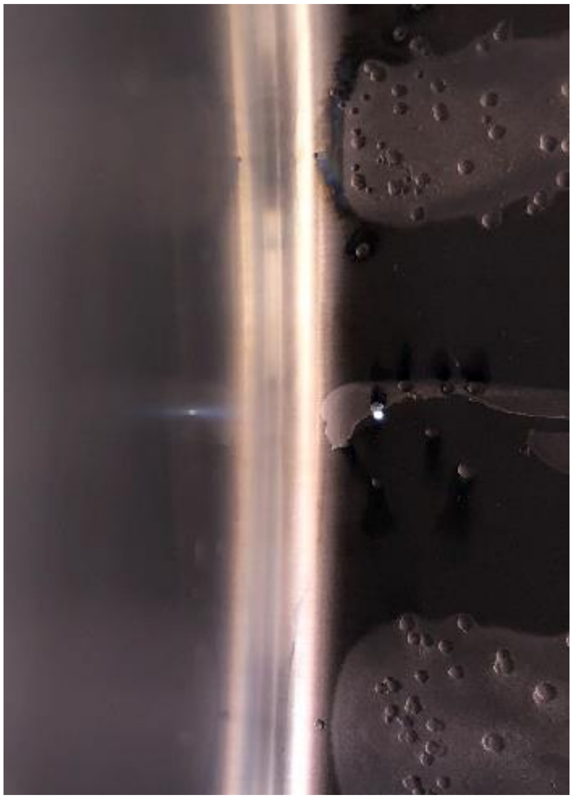


FIGURE 8 VISIBLE HOLE FROM FULL PENETRATION PIT.

FIGURE 9 INNER SURFACES OF WHOLE SAVY AFTER BEING PITTED.

3.5 Drop testing results of pitted SAVY

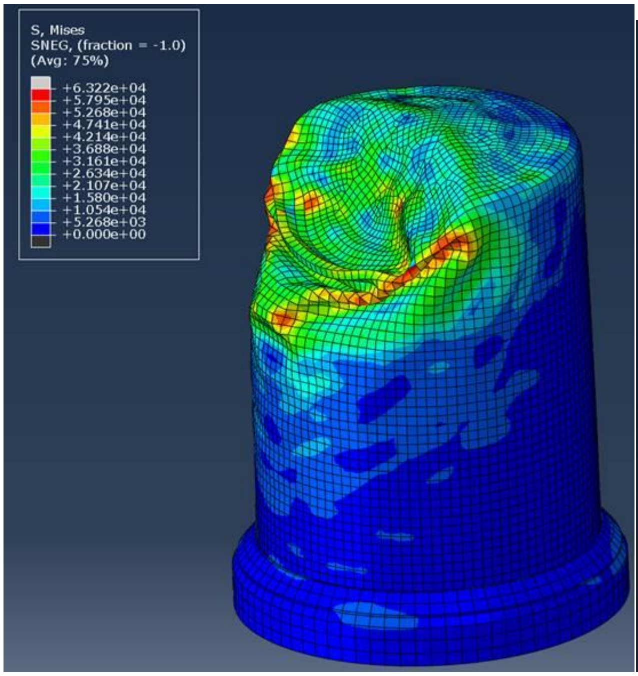
The helium leak tester in PF-4 was used on the corroded SAVY container before it was loaded with the payload in order to get a pre-drop helium leak rate and to ensure that none of the pits that were generated corroded through the thickness of the container. The pre-drop He leak rate was 7.9 x 10^{-8} atm cc/sec. The container was then taken to the drop tower at TA-03 Building 130. The container was visually inspected to identify the inner bottom radius region with the highest density of pitting which was the target impact location (Figure 10). The payload was made up to be 15.0178 kg bringing the total gross weight of the corroded container to 18.3742 kg. The loaded corroded container was then rigged in a sling and hung by the drop mechanism as was seen in Figure 2. The container was dropped from 12 feet and there was no evidence of failure (Figure 11). The container was removed from the drop tower and the payload was removed from the dropped container. A post-drop helium leak rate was obtained by taking the now empty SAVY back to PF-4. The helium leak rate was recorded as 1.2 x 10^{-7} atm cc/sec. There was a 51.9% increase in helium leak rate between the re and post drop rates. The increased helium leak rate can likely be explained by an increase in background helium in the system before the leak rate was measured. The background helium had a 191.2% increase in background from pre to post helium leak rate measurements.

FIGURE 10 CORRODED REGION INSIDE OF SAVY WITH THE HIGHEST DENSITY OF PITS.

FIGURE 11 DAMAGE POST-DROP TEST WITH BLACK "X" INDICATING TARGET REGION.

4. Discussion

The intent of this study was to identify what size a pit would need to be to cause a failure so that a lifetime prediction could be made at the current pit growth rates. The approach that was used to aid in the identification of the critical pit size was to understand the effect that different pits have on the mechanical properties and apply those degraded properties to a finite element analysis model. Then a corroded container was dropped and a model with the corresponding predicted mechanical properties was run. Comparing these two results (Figure 7 and Figure 11), with respect to deformations, shows that the FEA model was significantly more conservative with respect to the amount that the pits reduced the strength of the container body. It is believed that the reason that the model did not agree with the actual drop test on the pitted container due to the mechanical property data from the micro-tensile test apparatus were not truly representative of a container that has a distribution of pits. The pits that were observed in the dog bone samples that were used in the micro-tensile tests crossed the entire gauge width. To improve the models accuracy tensile tests should be completed on samples of another container that has been exposed to identical conditions as those used for the container that was dropped. These new data can be used in the FEA model and it is expected there will be a higher degree of agreement between the FEA model and the physical deformation seen in the drop.



Although the true critical pit size was not identified in this study to this point, it is worth highlighting the fact that the container that was dropped included pits that were between 35% and 55% through the thickness of the wall. This is an exceptional result and demonstrates that the SAVY-4000 is capable of undergoing a considerable degree of corrosion without failing in a drop scenario.

5. Conclusion

The primary conclusion that can be made from this study is that pits with depths of between 35% and 55 % that are relatively evenly distributed throughout a 5 quart SAVY-4000 will not compromise the ability to withstand a drop that is equivalent to the center-of-gravity over bottom corner design qualification drop test. To better understand what size a pit will need to be to cause a failure in the worst postulated drop scenario it will be necessary to measure the actual mechanical properties in a container with an even distribution of pits that are the same sizes as those in the container that was dropped. In order to extended the lifetime beyond the current 15 years it will be critical to have a method of predicting the rate at which pits grow when placed in the harshest storage conditions as well as understanding the size of a pit that will cause a failure during a drop.

6. References

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